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PHYSICOCHEMICAL PARAMETERS AND IODINE ENRICHMENT OF ROCK SALT FROM KEANA LGA

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Abstract

This study deals with the determination of the physicochemical parameters and iodine enrichment of rock salt mined in Keana Local Government of Nasarawa State. Five samples were collected from different pot spots and analysed for their physicochemical parameters as well as seeking cost effective ways of enriching the rock salt with iodine to help combat cases of goitre and brain retardation which are iodine deficiency diseases. The physicochemical parameters: pH, alkalinity, conductivity, total dissolved solids (TDS) and total suspended solids (TSS), were determined in the rock salt. The values ranged as follows: pH (6.0 – 8.2), conductivity (10.35 – 10.340□S/cm), alkalinity (0.01 – 0.04mg/L), total dissolved solids (1570 – 1583mg/L) and total suspended solids (0.03 – 0.19mg/L). The results of the physicochemical parameters where all below the permissible limit except for TDS which was higher than the permissible limit. The rock salt was also enriched with potassium iodate using the spray (wet) method and dry method. The result of the iodometric titration shows that rock salts were enriched with iodine. Using the spray method, iodine levels were within the range 75.15 – 80.14ppm, while salts enriched using dry method indicated that iodine levels were within the range 34.27 – 38.94ppm. The result shows that iodine enrichment using the spray method is more effective in attaining recommended level of iodine in rock salt at the point of production.

Key words: Keana, iodine, rock salt, potassium iodate, physicochemical parameters, phenolphthalein.

Introduction

Salt is a basic household condiment as well as a source of chlorine, whose importance cannot be over-emphasized. However, in terms of domestic consumption, WHO has mandated that salt meant for human consumption should be fortified with iodine to help combat health cases of goitre: a disease of the thyroid gland as well as cases of mental retardation.

The Nigerian economy is dwindling and as such needs to look inwards to boost her economy. One of such is taking advantage of the multi-billion-naira salt deposit within her jurisdiction. However, to achieve this, cost effective and efficient ways to mine salt and enrich it with iodine in accordance with World Health Organization (WHO, 2014) recommendation needs to be addressed.

Salts have been found to be mined locally in Keana Local Government of Nasarawa State in Nigeria at a very small scale, however this salt has little or no iodine content due to factors ranging from lack of awareness on the part of the small-scale manufacturers, to cost efficient ways of enriching the salt with iodine on the part of enlightened manufacturers. Thus, this study looks into determining the physicochemical parameters of locally mined salt and ways or methods of enriching locally mined salt in Keana Local Government in Nasarawa State with iodine.

Materials and Methods

Materials

The following reagents were used: Sodium thiosulfate solution (0.005M), 1% starch solution, Distilled water, 10% Potassium Iodide (KI), 1M Sulfuric acid (H_2SO_4), Potassium Iodate (KIO_3), Phenolphthalein and methyl orange, and Bromocresol green.

Sample Collection and Sample Treatment

Five samples were collected from different pot spots around the sample area as shown in Table 1, using clean stainless-steel containers. It was thoroughly mixed and transferred into clean and labelled polythene bag for laboratory analysis.

The salt sample were dried at room temperature for about 6hours and sieved through 2.00mm mesh to obtain a representative sample.

Table 1: Location of Study Samples

Sample	Sample Spots
SS1	Opu kh'aminye (Aminye spot)
SS2	Opu kh'ilikpi (Ilikpi's spot)
SS3	Opu khonyawole (Onyawole's spot)
SS4	Opu kh'abashe (Abashe's spot)
SS5	Opu kho'oweyin. (Oweyin spot)

Determination of Iodine by Iodometric Titration

The iodine content in salt samples was determined using the iodometric titration method. 50g of the salt sample is weighed and dissolved in 250 mL of distilled water. Once the salt dissolved, 2 mL of H_2SO_4 and 5 mL of KI was added to 50 mL of the aliquot of salt solution. This will turn yellow in the presence of iodine. The reaction mixture was then kept in a dark place (with no exposure to light) for 10 minutes to reach the optimal reaction

time, before titrated with sodium thiosulfate using starch (2 mL) as the indirect indicator. The concentration of iodine in salt was calculated based on the titrated volume (burette reading) of sodium thiosulfate according to the formula mentioned below:

Calculation:

mg/kg (ppm) iodine =

$$\frac{\text{titration volume (mL)} \times 21.222 \times \text{Normality of sodium thiosulfate} \times \text{initial volume (mL)}}{\text{salt sample weight (Kg)} \times \text{Used volume (mL)}}$$

Physicochemical Parameters

Alkalinity

A volume of the sample dissolved in distilled water, and H_2SO_4 titration cartridge corresponding to the expected alkalinity concentration as mg/L $CaCO_3$ was selected. A clean delivery tube was then inserted into the titration cartridge and the cartridge attached to the titration body. The delivery knob was turned to eject a few drops of titrant, then the counter was reset to zero and the tip wiped. Using a graduated pipette, 10 mL of sample solution was measured and transferred into a 250 mL conical flask. Diluted to 100 mL mark and 4 drops of phenolphthalein indicator was added and swirled to mix. When the solution turns pink, it was then titrated to colourless endpoint. The delivery tube tip of the titrant was placed into the solution and the flask swirled while titrating with H_2SO_4. The number of drops required was then recorded.

Calculate:

$$\text{Phenolphthalein Alkalinity (PA) mg/L } CaCO_3 = \text{Digits} \times \text{Digits multiplier (10)}$$

3 drops of bromocresol green – methyl red indicator was added and swirl to mix. Then continued titrating with H_2SO_4 to a light pink colour. The number of digits was then recorded.

Calculate:

$$\text{Total Alkalinity (TA) mg/L } CaCO_3 = \text{total digits} \times \text{digit multiplier}$$

(Jamal, 2017).

Total Suspended Solid

This was performed using gravimetric method. 5g of the sample was weighed and dissolved in distilled water in a beaker. The filter paper was dried in the oven for 20 mins at 105ºC. This was then cooled, and its weight measured and recorded. This was repeated until a constant weight was obtained between two successive readings. The sample solution was stirred with a glass rod and 20mL of the sample was measured quickly and filtered through the dried filter paper into a beaker. The filter paper + residue was then dried in the oven at 105ºC, cooled in the desiccator to balance temperature and weighed. It was reweighed again until a constant weight was obtained.

Calculation:

$$\text{mg total suspended solids/L} = \frac{(A-B) \times 1000}{\text{Sample volume in mL}}$$

Where A = weight of filter paper + dried residue, mg and B = weight of filter paper, mg

Total Dissolved Solids

This was performed using gravimetric method. 5g of the sample was weighed and dissolved in distilled water in a beaker, and the sample solution was then filtered. A beaker was dried in an oven for 15 mins at 105ºC, and the weight was measured quickly and recorded using a weighing balance. This step is repeated until a constant weight was obtained for two successive readings, and the final weight of the dry beaker was recorded. The sample solution was stirred and 20mL was measured into the dry beaker and evaporated to dryness in the oven at 180ºC, and then cooled in the desiccator. The weight of beaker and dry solid was then recorded, reweighed again until a constant weight is obtained.

Calculation:

$$\text{mg Total Dissolved Solids/L} = \frac{(A-B) \times 1000}{\text{Sample volume in mL}}$$

Where A = weight of dried residue + crucible mg and B = weight of dish mg

(Total Dissolved Solids (TDS), Water & Conductivity, 2017).

Conductivity Test

This test was carried out using a conductivity meter. Conductivity was measured with a probe and a meter. Voltage was applied between two electrodes in probe immersed in the sample water which contains 5g of sample dissolved with distilled water in a 100cm^3 volumetric flask. The drop in voltage caused by the resistance of the water was used to calculate the conductivity per centimetre. After a while the meter displayed the conductivity of the solution and the measurement was then recorded (PharmaSky, 2020).

pH Test

This was carried out using standard method recommended by Brikowski (2003).

Enrichment of Salt with Iodine

The salt sample was first dissolved in water and then filtered, to remove dirt and other suspended particles. The filtrate was then evaporated to dryness and then crushed to ensure evenness of salt particles.

Enrichment of salt with iodine using the dry mixing method was done when 84mg of KIO_3 was then measured and added to 1kg of the evaporated dry salt sample slowly while the dry salt was being mixed in a bowl.

Enrichment of salt with iodine using manual spraying of the salt was done when 1g of KIO_3 was measured and dissolved in 30mL of distilled water. 2.5 mL of the KIO_3 solution was then measured and diluted to 10mL in a spraying bottle. The resulting solution was then spray mixed by hand with 1kg of salt (IGN, 2020).

Results and Discussion

Table 2: Result of Physicochemical Parameters of Rock Salt Sample

PARAMETERS	SS1	SS2	SS3	SS4	SS5	WHO STANDARDS
pH at 10 % Solution	6.0±0.02	7.1±0.32	7.0±0.22	8.2±0.38	8.0±0.09	6.5-8.5
Conductivity 2)	10.39±1.02	10.35±2.12	10.36±1.87	10.40±1.46	10.38±2.40	300±1.32
Total Dissolved Solid (mg/L)	1578±12.0	1572±16.3	1570±22.6	1583±18.0	1574±20.2	500±13.6
Total Suspended Solid (mg/L)	0.19±0.002	0.04±	0.03±0.004	0.14±0.006	0.11±0.009	500
Alkanility (mg/L $CaCO_3$)	0.01±0.004	0.02±0.006	0.02±0.007	0.04±0.001	0.04±0.002	120

SS1 - Opu kh'aminye (Aminye spot) SS4 - Opu kh'abashe (Abashe's spot)

SS2 - Opu kh'ilikpi (Ilikpi's spot) SS5 - Opu kho'oweyin. (Oweyin spot)

SS3 - Opu khonyawole (Onyawole's spot)

Table 3: Result of Iodine Content of Rock Salt Sample

	SS1	SS2	SS3	SS4	SS5
Wet (ppm)	75.15±1.20	79.89±2.34	80.42±4.02	80.11±1.28	78.83±0.38
Dry (ppm)	34.27±1.24	38.73±2.10	36.08±1.80	38.94±1.76	37.46±1.56
WHO/UNICEF/ICCIDD recommended levels at production level (ppm)	50-80	50-80	50-80	50-80	50-80
WHO/UNICEF/ICCIDD recommended level at household level (ppm)	20-50	20-50	20-50	20-50	20-50

SS1 - Opu kh'aminye (Aminye spot) SS4 - Opu kh'abashe (Abashe's spot)

SS2 - Opu kh'ilikpi (Ilikpi's spot) SS5 - Opu kho'oweyin. (Oweyin spot)

SS3 - Opu khonyawole (Onyawole's spot)

Table 4: Result of Physicochemical Parameters of Rock Salt Sample After Enrichment with Spraying Method

PARAMETERS	SS1	SS2	SS3	SS4	SS5	WHO STANDARDS
pH at 10 % Solution	6.0	7.1	7.0	8.2	8.0	6.5-8.5
Conductivity (µS/cm)	35.31	37.11	41.38	41.29	36.88	300
Total Dissolved Solid (mg/L)	1578	1572	1570	1583	1574	500
Total Suspended Solid (mg/L)	0.19	0.04	0.03	0.14	0.11	500
Alkanility (mg/L $CaCO_3$)	0.01	0.02	0.02	0.04	0.04	120

Table 5: Result of Physicochemical Parameters of Rock Salt Sample After Enrichment with Dry Method

PARAMETERS	SS1	SS2	SS3	SS4	SS5	WHO STANDARDS
pH at 10 % Solution	6.1	7.1	7.0	8.2	8.0	6.5-8.5
Conductivity (µS/cm)	17.51	19.67	18.36	19.89	18.90	300
Total Dissolved Solid (mg/L)	1575	1571	1572	1579	1574	500
Total Suspended Solid (mg/L)	0.18	0.05	0.03	0.17	0.13	500
Alkanility (mg/L $CaCO_3$)	0.15	0.02	0.02	0.04	0.04	120

Rock salt samples from Keana was also analysed for physicochemical parameters which include; pH, electrical conductivity, total dissolved solids (TDS), total suspended solids (TSS) and alkalinity and results are shown in Table 2.

The pH rock salt sample when dissolved in water for SS1 was 6.0 which is slightly acidic, while the other samples were alkaline with pH 7.1, 7.0, 8.2 and 8.0 for SS2, SS3, SS4 and SS5 respectively. The samples; SS2, SS3, SS4 and SS5 has their pH fall within the WHO recommended standards from 6.5 – 8.5 while SS1 fall below this standard level. Similar study was carried out to determine the pH of salt lake in Keana and was found to be 7.563±0.009 which is within WHO permissible limit of 6.5 -8.5 (Ogah, 2020).

The electrical conductivity of rock salt samples analysed where considerably low. The electrical conductivity values ranges from 10.35 – 10.39 □s/cm which are far below the WHO guidelines for conductivity, 300□s/cm. The conductivity of analysed samples were 10.39□s/cm, 10.35□s/cm, 10.36□s/cm, 10.40□s/cm and 10.38□s/cm for samples SS1, SS2, SS3, SS4, and SS5 respectively. When salts are dissolved in water, they separate into ions. Therefore, the higher the conductivity, the more likely the water has high concentrations of ions.

The TDS values of analyzed salt ranged from 1570–1583mg/L, which are far above the WHO guideline value of 500 mg/L. The TDS of analysed samples were 1578mg/L, 1572mg/L, 1570mg/L, 1583mg/L and 1574mg/L for samples SS1, SS2, SS3, SS4, and SS5 respectively. High values of TDS can affect the taste, colour and odour of salt water. Similarly, TDS concentration level of about 746 mg/L which is above WHO limit, have been determined from analyzing underground water samples in Salt Lake City, India. Furthermore, higher concentrations of TDS have also been reported in Uganda, where levels of up to 27,000 mg/L were determined from analysis of Lake Katwe water samples (Banerji, 2017).
The TSS values were 0.19mg/L, 0.04mg/L, 0.03mg/L, 0.14mg/L and 0.11mg/L for samples SS1, SS2, SS3, SS4, and SS5 respectively. These TSS values are far below the WHO permissible level of 500mg/L. The values for alkalinity are also recorded. They ranged from 0.01 – 0.04mg/L which are safe below the WHO permissible level of 120mg/L.

Iodine levels of enriched rock salt samples was determine using classical methods of analysis i.e iodometric titration, and the resulting iodine level was calculated for each sample and shown in Table 3. Iodine levels for salt sample enriched using spraying or wet method was found to be within the range of 75.15 – 80.42 ppm. SS1 has the least iodine level of 75.15ppm, SS5 has 78.83ppm, SS2 has 79.89ppm, SS4 has 80.11pm while SS3 has the highest iodine level of 80.42ppm. All iodine levels fall within the WHO recommended levels of iodine of rock salt within the range of 50ppm – 80ppm at the manufacturing level.

Iodine levels of salt sample enriched using dry method was found to be within the range of 34.27 – 38.95ppm. SS1 has the least iodine level of 34.27ppm, SS3 has 36.08ppm, SS5 has 37.45ppm, SS2 has 38.73pm while SS4 has the highest iodine level of 38.94ppm. All iodine levels fall below the WHO recommended levels of iodine of rock salt within the range of 50ppm – 80ppm at the manufacturing level.

Rock salt samples enriched with iodine using the spraying method tended to be more efficient than that of dry method and achieving homogeneity of salt sample while mixing. According to IGN (2020), iodine levels of enriched salt tend to decrease by almost 50% by the time it gets to the households and also before consumptions. This decrease could be as a result of poor packaging at the point of manufacturing, storage time, and climate. With all these factors in view, iodine level of salt enriched using dry method would fall below the WHO recommended level of 20 -50ppm at household level, while those enriched using the spraying method will tend to be within the recommended level at the household level.

A comparison of the physicochemical parameters pre and post enrichment using spraying and dry method, indicates that enrichment by either dry or wet method has no significant difference on the parameters at P=0.05 probability.

Conclusion

In this study, the concentration of the investigated physicochemical parameters in rock salt samples were within the WHO guidelines except for TDS, even though it has no direct impact on consumers. Enrichment of rock salt with iodine using the spraying method proves to be more efficient among small salt producers than the dry method. Also, small or local salt producers should be sensitized on the importance of enriching their locally made salt with Iodine before it is sold out to household for consumption.

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